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## Study on the Nano and Micro Surface Morphology Effects on Interfacial Strength of Adhesively Bonded Bimaterials

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### Abstract

Adhesive joints have been used widely in various engineering applications. Many previous researchers reported that the adhesive joints with rough adherend surfaces result in improved adhesion strength. In this paper, nano and micro scale surface treatment effects on adhesion strength improvement for CFRP/aluminum interfaces were investigated. Aluminum specimens were chemically etched to fabricate micro-line patterns using the process of photolithography and then anodized to form nano scale surface morphology of an aluminum oxide layer across the whole patterned area. These nano size porous surface structure by anodizing allows the adhesive to fill the cavities and form new adhesive surface at the interface as this bi-material specimen was co-cured in the autoclave. By using these surface treated specimens, adhesion strength was investigated by comparing the maximum load bearing values in the single-leg bending tests. The experimental results showed that adhesion strength could be enhanced by applying the nano and micro scale surface morphology obtained from surface treatment on the metallic adherend. Mechanical interlock effect by micro scale pattern can be increased by nano scale surface morphology effect promoting additional adhesion strength at the interfaces.

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Key words : surface morphology, mechanical interlocking, mixed-mode, interfacial fracture toughness, single-leg bending test

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### 1. Introduction

Adhesive bonding is very important technique that adhesively joins two materials together by using an adhesive so that the surfaces of two adherends can be bonded permanently by forming chemical adhesion [1,2]. Since the 1950s, adhesive-bonded joints have been used widely in aircraft structures. Many part of surface area of transport aircrafts are made up of various types of honeycomb structures that adopt

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adhesive-bonding methods [2]. Today, adhesive bonding technology is considered to be one of the most important joining method in many engineering applications due to its advantageous characteristics over traditional mechanical joining methods, such as bolting, riveting, and welding.

Adhesive joints show us many advantages compared to traditional joining. The most significant advantage is the ability to have uniform stress distribution at the joints due to the large area involved in the joint geometry. Absence of holes, used in bolting and riveting, prevents undesirable stress concentration that could result in failure of the joint. Furthermore, adhesive bonding could be used to join actually all combinations of materials, since, unlike in welding, high temperatures are usually not necessary to create a joint [1]. It is also said that adhesive joints have good fatigue and damping properties. [1-3].

In order to ensure reliability in adhesive joints, efforts have been made to develop adhesion strength enhancement techniques. One of the most studied methods is to correlate the joint strength with surface roughness of the interface. It is generally known that surface roughness could improve adhesion strength due to mechanical interlocking effect induced by the protrusions of the bonding interface. However, recent study found that surface roughness is only an indirect parameter that contributes adhesion strength improvement while the area fraction of cohesive failure may give rather direct correlation to adhesion strength [4].

In the fabrication of aircraft structures, the morphology of the surface oxide on the metal was used to promote the integrity and long-term durability of metal/polymer bonds. They found that aluminum anodization pretreatment process produces oxide films on the metal surfaces which assist mechanical and chemical characteristics for bond durability [5].

In this study, aluminum was chemically etched to fabricate micro line patterns using the process of photolithography and then anodized to form nano scale surface morphology of an aluminum oxide layer across the whole patterned area. The effects of different scale surface morphology on adhesion strength improvement for CFRP/aluminum bi-material interfaces were investigated by comparing the maximum load bearing values by using the single-leg bending (SLB) tests.

## 2. Specimen Fabrication and Experimental Methods

### 2.1. Micro Scale Line Pattern Fabrication

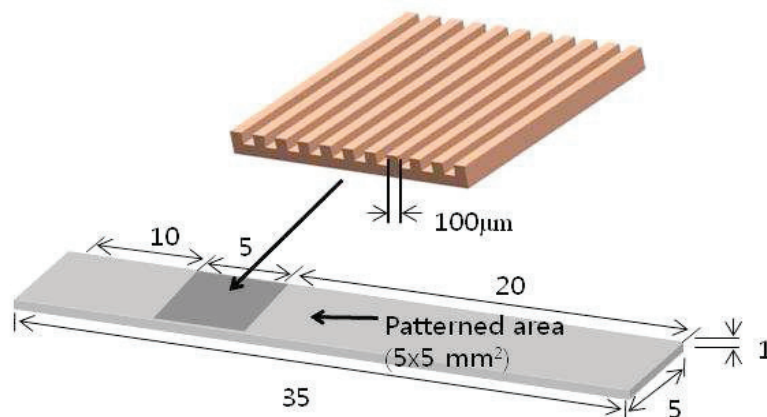


Figure 1. Specimen size and location of the line pattern

Micro scale line pattern was made on the aluminum surface in order to investigate surface morphology effects on adhesion strength improvement. Aluminum plate (Al7075) was cut into  $35 \times 5 \times 1 \text{ mm}^3$  pieces and finely polished using a rotating disk type polisher. The polished aluminum surfaces were spin-coated with a commercial photoresist (SU-8 from MicroChem, USA) and exposed to UV I-line (365 nm wavelength) through a transparency-printed mask. Using this conventional photolithography, a line pattern with a line width of  $100 \text{ }\mu\text{m}$ , as in the mask, was copied to the photoresist coating on the aluminum adherend by solidifying only the UV-exposed region. The geometry of the specimen and the location of the line pattern are illustrated in Figure 1. Then, the photoresist left on the substrate was used as a mask again in wet etching. The specimen was immersed into the etchant of the composition shown in Table 1. Regions in the aluminum surface not covered by the photoresist films were chemically etched to fabricate a micro scale line pattern on the aluminum adherend. The temperature was maintained at  $45^\circ$  during the etching.

Table 1. Etchant composition by percentages

Composition	Percentages (%)
$\text{H}_2\text{PO}_4$	80
$\text{HNO}_3$	5
$\text{CH}_3\text{COOH}$	5
Deionized water	10

## 2.2. Fabrication of Nano Scale Porous Surface Morphology



Figure 2. Sequence of anodizing to form an alumina layer with uniform pores

To get the nano scale porous surface morphology on the micro line patterned aluminum surface, the specimen was anodized to form a layer of alumina ( $\text{Al}_2\text{O}_3$ ) porous structure on the aluminum surface after the etching process. This anodizing process consists of the first and second formation of alumina layer in order to create uniform porous morphology across the whole line pattern region on the aluminum specimen. The alumina layer formed from the first anodizing process was intentionally removed using phosphoric acid and  $\text{CrO}_3$  since this additional step appears to give better surface pre-condition for the following second anodizing. (Figure 2) When the second anodizing was completed, an alumina layer, which is a pore-like structure with the pore diameter of about  $100 \text{ nm}$ , was successfully obtained.

The first anodizing was conducted at the voltage of  $40 \text{ V}$  and the temperature of below  $0^\circ$ . The alumina removal was carried out at  $70^\circ$  and the voltage supplied for the second anodizing was elevated to  $100 \text{ V}$ , more than twice of that from the first anodizing, to ensure that the pores are big enough for the epoxy to penetrate into it afterwards in bonding process.

The micro scale pattern geometry and the nano scale surface morphology could be observed by SEM equipment. As shown in Figure 3.(a), micro-scale line pattern on the aluminum adherend was observed clearly. Due to the isotropic characteristic of wet etching process, we can observe that the cross sections of the line patterns were cut down to be semi-circular. The excavated and preserved line width ratio can be determined by the amount of undercut that have taken place on the specimen during the etching. Therefore, by varying the etching time, it is possible to control the excavated and preserved line pattern width ratio. And for the nano scale morphology, the diameter of approximately 100 nm of pore morphology was attained across the whole patterned area as shown in Figure 3.(b).

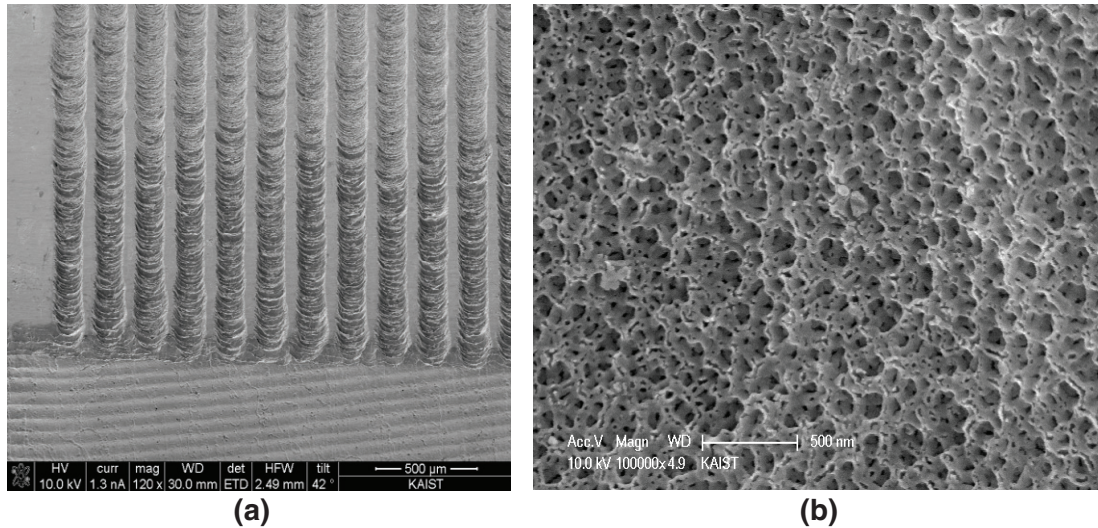


Figure 3. SEM images of (a) micro scale line pattern and (b) nano scale surface morphology

### 2.3. SLB Specimen Fabrication and Static Strength Measurement

The bi-material SLB specimen was fabricated using the co-cure bonding method. First, CFRP prepregs (USN 150B, SK chemicals) were cut into 40×5 mm<sup>2</sup> and stacked to each other to make up desired thickness. Then the CFRP adherend was pre-bonded to the aluminum adherend before curing. Next the specimen was put into the autoclave and went through the standard curing process of the CFRP prepregs. During the curing process of the composite material, bonding process between composite and aluminum adherends is achieved simultaneously by the excess resin spreading on the surface of the aluminum adherend. Then, three point bending tests were conducted using the SLB specimens. Load and displacement data were recorded while the jig in the middle was moving downward 10 μm/s, causing fracture of the specimen.

To apply a mixed mode loading conditions to the bi-materials adhesive specimen, The SLB test configuration was considered, and three point bending tests were conducted as shown in Figure 4.

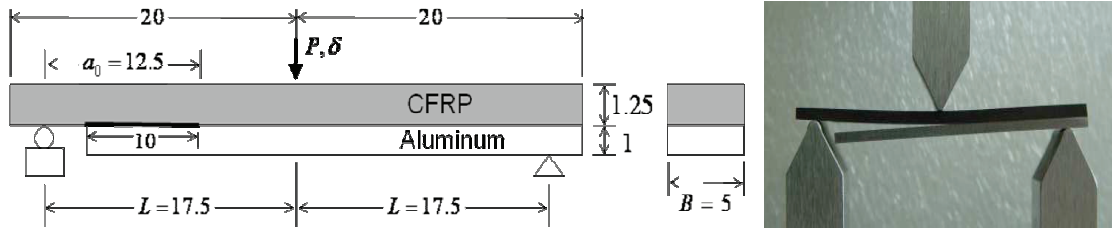


Figure 4. CFRP/aluminum bonded SLB specimen

### 3. Results

To investigate the effect of the nano and micro scaled interfacial morphology on the adhesion strength, Specimens of different surface treatment were prepared. The basic type specimens are consisted of SLB specimens with only sandpaper-abraded aluminum adherends, relatively easy and common surface pre-treatment used to create moderate surface roughness. The second type is only micro scale line patterned specimens and third type is micro line patterned with anodizing process to form nano scale surface morphology.

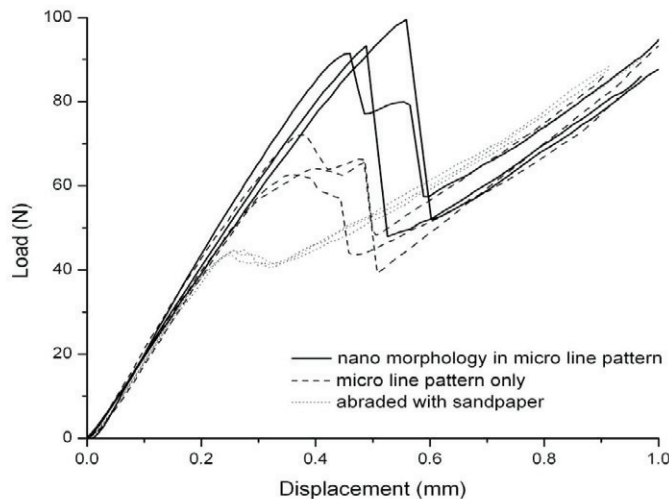


Figure 5. Load-displacement curves under three different adherend surface conditions

The load-displacement curves of SLB tests under the three different adherend surface conditions are shown in Figure 5. The load bearing capacity varies with different surface conditions while the loads increase almost linearly before they reach the maximum values. It is observed that the SLB specimens with nano scale morphology in micro scale line pattern resulted in the highest maximum load bearing capacity. The SLB specimens with only micro scale line pattern exhibited the second highest in load bearing capacity among the three groups of surface conditions. Abraded adherends only resulted in the lowest load bearing capacity with just above 40 N. Each test with the same surface condition was repeated three times to confirm that the results are reproduced within a certain deviation.



#### 4. Discussion

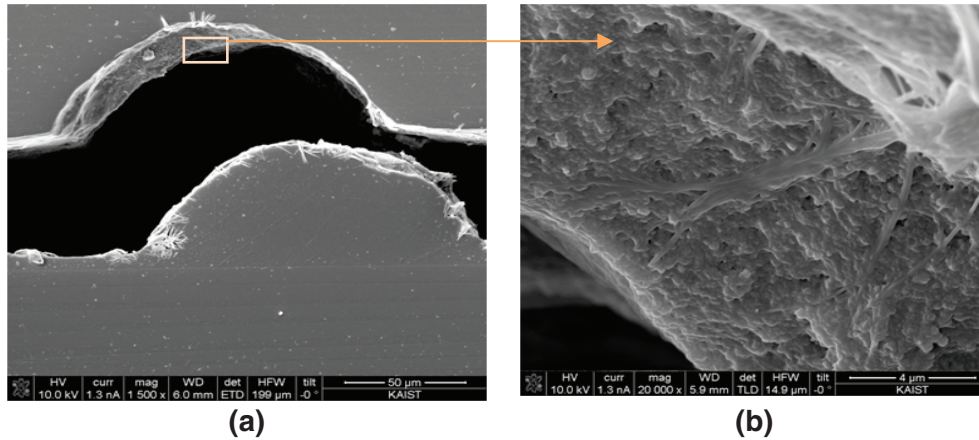


Figure 6. SEM images of fracture in (a) micro scale line pattern interface and (b) aluminum surface

The experimental result was analyzed by using the SEM images of the fractured interface of specimens as shown in Figure 6. Unlike in pure sliding mode (mode II), it looks like macroscopically that the micro scale line pattern of epoxy, which is low part of Figure 6 (a), of interface of specimens remains intact when opening and sliding mode coexist, which suggests that the micro scale line pattern did not provide sufficient mechanical interlock effect when fractured under this mixed mode test (Figure 6 (a)). However, the magnified image of the fractured interface to check more microscopically show that the nano scale aluminum oxide layer is covered with epoxy. This means that this nano scale porous morphology effectively captured epoxy, exhibiting nano scale mechanical interlock effect across the whole interface (Figure 6.(b)). This cohesive failure mode induced by nano scale porous surface morphology consumed more energy for fracture propagation and thus ultimately increased the load bearing capacity of the interface of CFRP/aluminum bi-material under the mixed mode loading condition.

#### 5. Acknowledgements

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